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Professor Jian-Ku Shang

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14. ABSTRACT

Thermosetting polymers, used in a wide variety of applications ranging from microelectronics to composite airplane wings, are susceptible to damge in the form of cracking. Often these cracks form deep within the structure where detection is difficult and repair is virtually impossible. Regardless of the application, once cracks have formed within polymeric materials, the integrity of the structure is significantly compromised. Inspired by biological systems in which damage triggers a healing response, this project focuses on the development of a new structural polymeric material with the ability to autonomically heal cracks. This work will lead to safer and more reliable materials in a wide range of applications and represents the first step in developing materials systems that possess greatly extended lifetimes.

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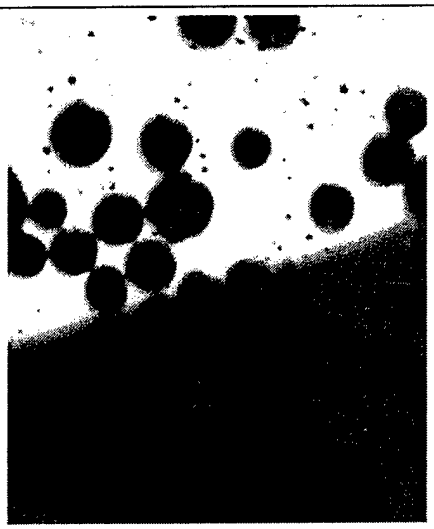
DEVELOPMENT OF SELF-HEALING STRUCTURAL COMPOSITE MATERIALS

AFOSR GRANT # F49620-00-1-0094



Optical micrograph of self-healing epoxy.

A microencapsulated healing agent (red) is released from microcapsules as the crack tip moves through the material The crack front is positioned in the center of the image and the healing agent covers the fracture plane behind it. Once released, the healing agent contacts the chemical catalyst (black) suspended in the epoxy and polymerization occurs leading to rebonding of the fracture plane.

ABSTRACT

Thermosetting polymers, used in a wide variety of applications ranging from microelectronics to composite airplane wings, are susceptible to damage in the form of cracking. Often these cracks form deep within the structure where detection is difficult and repair is virtually impossible. Regardless of the application, once cracks have formed within polymeric materials, the integrity of the structure is significantly compromised. Inspired by biological systems in which damage triggers a healing response, this project focuses on the development of a new structural polymeric material with the ability to *autonomically* heal cracks. This work will lead to safer and more reliable materials in a wide range of applications and represents the first step in developing materials systems that possess greatly extended lifetimes.

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1. Objectives (Note: unchanged from proposal)

The objective of this research is to develop a new class of structural material capable of self-healing. We plan to limit our investigations to polymer composites and thermosetting matrices. Specifically, we will focus on epoxy and dicyclopentadiene (DCPD) matrix composites with glass or graphite reinforcing fibers, but will include an experimental screening of other potential matrix systems.

Material System Development

- experimentally screen a broad spectrum of potential systems
- demonstrate *in-situ* polymerization of healing agent
- optimize matrix/healing agent system based on model "crack" experiments

Experimental Assessment of Self-Healing

- demonstrate and measure extent of self-healing at three levels of complexity: neat resin specimens, "model" composites, high volume fraction composites
- measure the effect of microspheres on composite properties
- evaluate the environmental stability of self-healing composite systems

Modeling of Self-Healing Process

- use a computational mechanics tools to investigate:

– initiation of the healing process including microsphere rupture or debonding
– the development and extent of repaired "strength" in composite specimens

2. Status of Effort

In the first year ('99-'00) of our grant, *in situ* self-healing for a neat epoxy resin was successfully demonstrated. To achieve this objective many technical challenges were overcome including microencapsulation of the healing agent, stability of the catalyst and materials system, development of healing protocols, etc. The result was a self-healing material system capable of 70% recovery of virgin fracture toughness even after catastrophic failure - a remarkable level of recovery considering the nature of the damage we are attempting to heal. The impact of these ground breaking results, which were published in the Feb. 15, 2001 issue of *Nature,* is evidenced by the overwhelming international press coverage including the front page of the *Washington Post*, *US News&World Report*, *Wired Magazine*, *ABC World News Tonight*, *National Public Radio's Morning Edition*, *BBC Radio*, *National Geographic*, the *Discovery Channel* and numerous others. Over the second year ('00-'01), we successfully characterized the healing kinetics, analyzed the micromechanical behavior of the capsules, and extended our self-healing concept from the neat epoxy to a structural graphite-epoxy composite material. During the past year ('01-'02) our efforts have focused on optimizing the first generation healing system for improved healing efficiency, faster recovery, and lower concentration of additives. We have successfully demonstrated ca. 90% recovery at concentrations below 5% (wt) microcapsules. We have also shown accelerated kinetics of recovery with a new healing agent that yields better than 40x kinetic rates. We are currently exploring new chemistries and matrices, as well as new applications of self-healing technology, with particular emphasis on fatigue loading and retardation of crack growth rates.

3. Accomplishments

3.1 Advancements in healing agent chemistry

One of the objectives of our research over the past year has been to investigate new approaches to reducing the recovery time of self-healing epoxy. Dicyclopentadiene (DCPD), our current healing agent, can exist as an *endo* (**1**) or an *exo* (**2**) isomer. Because commercially available DCPD is >95% *endo*, most of the applications of DCPD involve the *endo* isomer. Previous studies on the ring-opening-metathesis-polymerization (ROMP) of other norbornene derivatives have shown that *exo* isomers often react faster than the corresponding *endo* isomers. No direct examinations of the difference in ROMP kinetics of *endo*- and *exo*-DCPD have previously been reported with a Ru based catalyst, as we use in our self-healing system, but a few studies suggest that *exo*-DCPD reacts differently than *endo*-DCPD. Consequently, it seemed likely that *exo*-DCPD would have higher ROMP rates than *endo*-DCPD using Grubbs' catalyst, and a deeper understanding of this behavior could prove valuable in the generation of new self-healing materials.

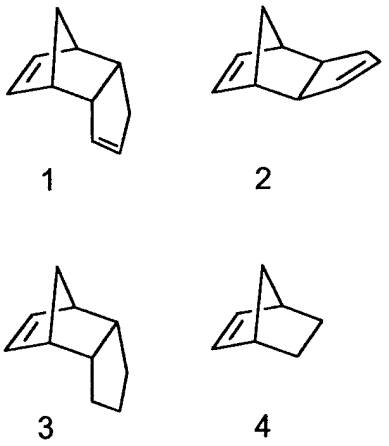

Fig. 1. Chemical structures of healing agents. (a) exo-DCPD. (b) endo-DCPD. (c) endo-1,2 dihydroDCPD. (d) norbornene.

Using in situ NMR, the *exo* isomer of DCPD was found to be more than an order of magnitude more reactive than the *endo* isomer. *endo*-DCP was found to have reactivity similar to its partially saturated counterpart **3**, suggesting that the cause of the rate difference between the two isomers of DCPD is primarily steric in nature. This interaction is shown to be entropic and is suspected to originate from an interaction of the penultimate repeat unit and the incoming monomer. Additionally, the alkylidene generated during the polymerization of *endo*-DCPD was found to form an intramolecular complex, but this complex only affects the rate slightly. We are currently investigating microencapsulation routes for *exo*-DCPD to facilitate self-healing trails in epoxy test samples.

3.2 Optimization of self-healing epoxies

Over the past year we have carried out a comprehensive investigation of the correlative fracture and healing mechanisms of the self-healing system. The effects of microcapsule concentration, catalyst concentration and microcapsule size were studied with a view towards optimizing healing efficiency (defined as the ability to recover fracture toughness). Healing efficiency was measured using a tapered double-cantilever beam

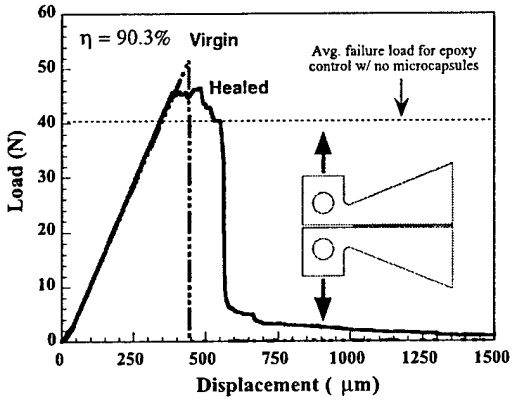

Fig. 2. Load-displacement data for a virgin and healed epoxy fracture toughness specimen.

(TDCB) fracture test protocol. Representative load-displacement data for one sample with 5 wt% of microcapsules and 2.5 wt% catalyst are plotted in Fig. 2.

Microcapsule Concentration – Fracture samples containing 0% to 25% by weight of microcapsules (ca. 180 μm diameter) were tested to failure, allowed to self-heal and then re-tested. Both the virgin and healed fracture toughness are summarized in Fig. 3 as a function of capsule concentration. The addition of microcapsules significantly improves the toughness of the neat epoxy. A maximum is achieved between 10-15 wt% capsule concentration. Observation of the fracture surface (Fig. 4) and the characteristic "tails" originating from the broken spheres revealed that crack pinning may be the operative toughening mechanism. This toughening mechanism has been widely reported for the addition of particulate fillers to thermosetting resins. Hackle patterns on the fracture surface indicate additional toughening due to localized shear deformation

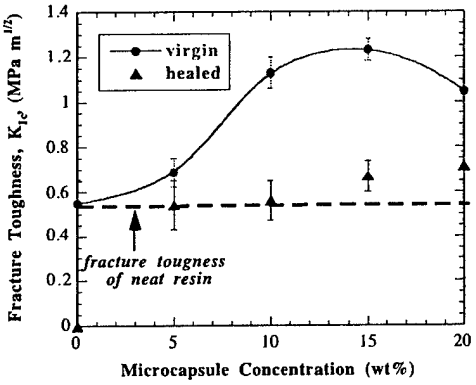

Fig. 3. Effect of microcapsule concentration on virgin fracture toughness.

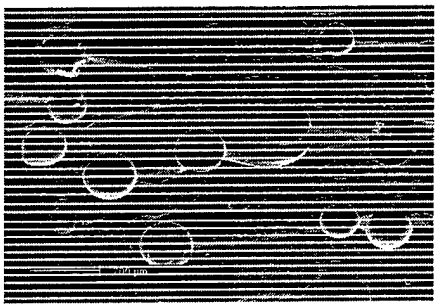

Fig. 4. ESEM image of crack plane for 10 wt% microcapsules.

In our previous investigations of this self-healing system, the microcapsule concentration was chosen to be 10 wt% to nearly maximize virgin fracture toughness and healing efficiencies ca. 70% were obtained. Our more recent data (Fig. 2) reveal that higher healing efficiencies can be obtained when the virgin fracture toughness is not maximized. By reducing the capsule concentration to 5% healing efficiencies of greater than 90% were obtained, such as for the sample data in Fig. 2.

Catalyst Concentration – The effect of the ratio of Grubbs catalyst to DCPD monomer was investigated by measuring the healing efficiency in samples with Grubbs catalyst concentration from 0 wt% to 4 wt%. The healed fracture toughness increased with the addition of more catalyst. However, the relative gain in healed fracture toughness actually decreased for each additional increment of catalyst concentration. For catalyst concentration beyond 3 wt%, the virgin fracture toughness decreased with further addition of catalyst. Although a high healing efficiency resulted at these high catalyst concentrations, gains were a result of degradation of the virgin properties.

Microcapsule Size – Self-healing fracture samples were fabricated with microcapsules ranging from 20 to 1000 μm in diameter. Microcapsule size was controlled by changing the agitation rate during the *in situ* polymerization encapsulation process. Healing efficiency remained nearly constant with decreasing microcapsule diameter down to about 20 microns. Below 20 microns, larger concentrations of capsules were required to deliver enough healing agent to the crack plane and maintain healing efficiency.

3.3 Self-healing structural composites

Transitioning these promising results from neat resins to structural (fiber-reinforced) composites is challenging. Previously, we were able to show that healing of delaminations could be *self-activated* by incorporating the same catalytic trigger within the matrix of a fiber-reinforced composite. In situ polymerization kinetics was shown to play a crucial role in determining the degree of repair achieved. Over the past year we have demonstrated a fully *self-healing* structural composite system that incorporates a microencapsulated healing agent and a catalytic trigger into the polymer matrix. The focus of our study was the repair of delamination damage in double cantilever beam specimens.

Two different types of controls were tested in addition to self-healing composites. Reference and self-activated specimens involve the injection of the monomer healing agent or a pre-catalyzed mixture of the healing agent and ROMP catalyst into the crack plane after an initial fracture. Seven sets of samples were analyzed: one set of reference specimens, one set of self-activated specimens, and five different sets of *in situ* specimens. The standard *in situ* specimen contained 20 wt% microcapsules in the resin that was used to impregnate the central four layers of the composite panel. The influence of capsule loading on healing efficiency was also investigated with samples of 10 wt% and 15 wt% microcapsule concentrations. One group of specimens was allowed to heal at an elevated temperature to measure the influence of healing temperature on the effectiveness of self-healing. While the healing efficiency for most of the specimens was determined after more than 48 hours from when the damage occurred, specimens from one batch were tested at nine different times after the virgin loading and damage initiation from 10 minutes to 48 hours.

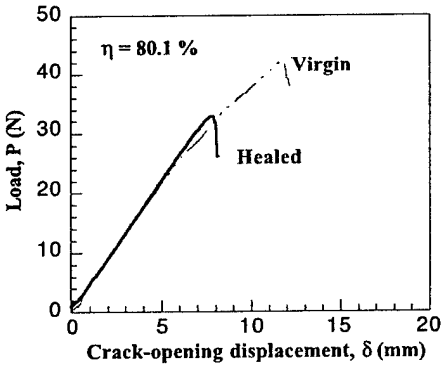

Fig 5. Load-displacement data for in situ healing of structural composite specimen. [healing @ 80°C for 1 h]

Fig. 6. ESEM image of the fracture plane of self-healing structural composite showing polymerized healing agent covering crack face.

The reference specimens established that the healing agent system is capable of achieving 100% repair in the composite material. The self-activated specimens demonstrated that the embedded catalyst is capable of polymerizing the healing agent and achieving high levels of repair (ca. 80%). The *in situ* specimens represent a fully integrated system that repairs the delamination autonomically.

After 48 hours of healing at room temperature the healing efficiencies for in situ specimens ranged from 38-47%. The reduced efficiency from the control groups appears to be due to retardation of the polymerization kinetics for composite specimens. Specimens which were allowed to heal at elevated temperature (Fig. 5) showed greatly increased healing efficiency ($\approx$ 80%). The effect of temperature is believed to be solely an increase in the kinetic rate of healing. As such, it appears that the polymerization kinetics

for high fiber volume fraction composites must be adjusted from that achieved in the neat resin in order to achieve optimal healing.

4. Personnel Supported

Faculty: S.R. White, J.S. Moore, P.H. Geubelle, N.R. Sottos, P.V. Braun
Graduate Students: J. Rule, E. Brown, M. Kessler, D. Therriault, S. Sriram, S. Viswanathan, A. Farag

5. Publications

Kessler, M.R. *Characterization and Performance of a Self-Healing Composite Material*, Ph.D. Dissertation, Department of Theoretical and Applied Mechanics, University of Illinois at Urbana-Champaign, 2002.

Kessler, M.R. and S.R. White, "Cure Kinetics of the Ring-Opening Metathesis Polymerization of Dicyclopentadiene," *Journal of Polymer Science Part A: Polymer Chemistry*, **40**:2373-2383, 2002.

Rule, J.D. and J.S. Moore, "ROMP Reactivity of *endo*- and *exo*-Dicyclopentadiene," *Macromolecules*, in press, 2002.

Brown, E.N., Sottos, N.R. and White, S.R., "Fracture testing of a self-healing polymer composite," *Experimental Mechanics*, in press, 2002.

Brown, E.N., Sottos, N.R. and White, S.R., "Microcapsule induce toughening in a self-healing polymer composite," to appear, Proceedings of the American Society of Composites, CD-ROM, Oct. 2002.

Brown, E.N., Sottos, N.R. and White, S.R., "Fracture testing of a self-healing polymer composite," Proceedings of Society of Experimental Mechanics XI International Congress on Experimental Mechanics, CD-ROM, 2002.

Brown, E.N., "Interdisciplinary Research: A Student's Perpsective," *Journal of Chemical Education*, 79:1 (2002).

White, S.R., N.R. Sottos, P.H. Geubelle, J.S. Moore, M.R. Kessler, S.R. Sriram, E.N. Brown, S. Viswanathan, "Autonomic Healing of Polymer Composite Materials," *Nature*, **409**:794-797, 2001.

Kessler, M.R. and S.R. White, "Self-Activated Healing of Delamination in Woven Composites," *Composites Part A: Applied Science and Manufacturing*, **32**:683-699, 2001.

Sriram, S. *Development of Self-Healing Polymer Composites and Photoinduced Ring Opening Metathesis Polymerization*, Ph.D. Dissertation, Department of Chemistry, University of Illinois Urbana-Champaign, 2001.

S.R. White, N.R. Sottos, J.S. Moore, and P.H. Geubelle, E.N. Brown, and M.R. Kessler "Autonomic Healing of Polymer Composites," Proceedings of the ASC 16th Technical Conference on Composite Materials, Sept. 9-12, 2001, Virginia Tech University, Blacksburg, VA.

S.R. White, N.R. Sottos, J.S. Moore, P.H. Geubelle, M.R. Kessler, S.R. Sriram, and E.N. Brown, "Self-Healing Polymer Composites," Proceedings of the 10th International Congress on Fracture, December 2-6, 2001, Honolulu, Hawaii.

Viswanathan, S. *Micromechanical Modeling of Self-Healing Polymeric Composites*, M.S. Thesis, Department of Aeronautical and Astronautical Engineering, University of Illinois Urbana-Champaign, 2000.

Brown, E.N. and Sottos, N.R., "Performance of embedded microspheres for self-healing polymer composites," Proceedings of Society of Experimental Mechanics IX International Congress on Experimental Mechanics, CD-ROM, 563-566, 2000.

Brown, E.N, Sottos, N.R., White, S.R., Geubelle, P.H., Moore, J., "Self-Healing Composites Using Embedded Microspheres: Fracture Toughness of Epoxy Matrix," Proceedings of ICTAM 2000, 20th International Congress of Applied Mechanics, p. 214, 2000.

6. Interactions/Transitions

6.1 Conference Presentations & Seminars

Fracture and Fatigue Behavior of a Self-Healing Polymer Composite, Brown, E.N., poster presentation, Materials Research Society Fall Meeting, Dec. 2-6, 2002, Boston, MA.

Fatigue Behavior of a Self-Healing Polymer Composite, Brown, E.N., Beckman Institute Nanohour, Beckman Institute, Nov. 20, 2002, Urbana, IL.

Development of a Self-Healing Polymer, Brown, E.N., Department of Physics and Chemistry Seminar, Chicago State University, Nov. 19, 2002, Chicago, IL.

Microcapsule-Induced Toughening in a Self-Healing Polymer Composite, Brown, E.N. American Society of Composites 17th Technical Conference on Composite Materials, Oct. 21-23, 2002, Purdue University, West Lafayette, IN.

Mimicking Biological Systems, a Self-Healing Polymer Composite, Brown, E.N., Mechanics and Materials Research Group Seminar, University of Wisconsin, Oct. 18, 2002, Madison, WI.

Autonomic Healing of Polymer Composites, White, S.R., and Sottos, N.R., Seminar given to Ciba-Geigy Corporation, Beckman Institute for Advanced Science and Technology, Sept. 23, 2002, Urbana, IL.

Autonomic Healing of Polymer Composites, White, S.R., Sottos, N.R., and Moore, J.S. Seminar given to Northrup-Grumman Corporation, Beckman Institute for Advanced Science and Technology, July 31, 2002, Urbana, IL.

Self-Healing Polymer Composites, White, S.R. Self-Healing Polymer Composites Technology Briefing, Beckman Institute for Advanced Science and Technology, July 23-24, 2002, Urbana, IL.

Self-Healing Polymer Composites, Brown, E.N., AFRL/MLBCO seminar, Wright-Patterson Air Force Base, July 15, 2002, Dayton, OH.

Self-Healing Composites, White, S.R. 14th U.S. National Congress on Theoretical and Applied Mechanics, June 23-28, 2002, Virginia Tech University, Blacksburg, VA.

Fracture Behavior of a Self-Healing Polymer Composite, Brown, E.N., 14th U.S. National Congress of Theoretical and Applied Mechanics, June 27, 2002, Blacksburg, VA.

Fracture Testing of a Self-Healing Polymer Composite, Brown, E.N. Society of Experimental Mechanics XI International Congress on Experimental Mechanics, June 10-12, 2002, Milwaukee, WI.

Autonomic Healing of Polymer Composites, White, S.R., Sottos, N.R., Geubelle, P.H. and Moore, J.S. Seminar given to Goodyear Corporation, Beckman Institute for Advanced Science and Technology, May 24, 2002, Urbana, IL.

Autonomic Healing of Polymer Composites, Sottos, N.R. 2002 R.L. McCullough Research Symposium, University of Delaware, May 22, 2002, Newark, DE.

Autonomic Healing of Polymers and Composites, White, S.R. IEEE-Aerospace 2002, March 11-15, 2002, Big Sky, MT.

Autonomic Healing of Polymer Composites, Sottos, N.R. Seminar given to Dept. of Chemical Engineering, Drexel University, Feb. 2, 2002, Philadelphia, PA.

Self Healing of a Polymer Composite Material, Brown, E.N., Department of Theoretical & Applied Mechanics Seminar, University of Illinois, Jan. 24, 2002, Urbana, IL.

Self-Healing Composites, White, S.R., Gordon Conference on Composites, Jan. 8-12, 2002, Ventura, CA.

Fracture Testing of a Self-Healing Polymer Composite, Brown, E.N. and Sottos, N.R., poster presentation, Gordon Conference on Composites, Jan. 9, 2002, Ventura, CA.

Self-Healing Polymer Composites, White, S.R. 10th International Congress on Fracture, December 2-6, 2001, Honolulu, Hawaii.

Autonomic Healing of Polymer Composites, White, S.R. Seminar given to Department of Mechanical Engineering, Washington University, Nov. 7, 2001, St. Louis, MO.

Autonomic Healing of Polymer Composites, White, S.R. Seminar given to Boeing Corporation, Nov. 7, 2001, St. Louis, MO.

Autonomic Healing of Polymer Composites, Sottos, N.R. American Society of Composites 16th Technical Conference on Composite Materials, Sept. 9-12, 2001, Virginia Tech University, Blacksburg, VA.

Autonomic Healing of Polymers, White, S.R. Seminar given to Jet Propulsion Laboratory, Pasadena, CA, August 23, 2001

Self-Healing Composites, White, S.R. Seminar given to Loctite Corporation, Hartford, CT, June 28, 2001.

Autonomic Healing of Polymeric Composites, White, S.R., Beckman Institute Information Technology Group Forum. Urbana, IL. June 14, 2001.

Autonomic Healing of Polymeric Composites, Sottos, N.R.. Society of Experimental Mechanics (SEM) Annual Conference. Portland, OR. June 4-6, 2001.

Self-Healing Composites, White, S.R. Seminar given to Institute of Materials Science, University of Connecticut, December 1, 2000, Storrs, CT.

Development of Self-Healing Polymer Composites, Sriram, S.R. MRS Fall Meeting. Boston, MA. Nov. 27-Dec. 1, 2000.

Self Healing Dielectric Layers in HDI Substrates, Brown, E.N. ASME International Mechanical Engineering Congress and Exposition. Orlando, FL. Nov. 5-10, 2000.

Materials Chemistry at the Interface of Science and Technology, Moore, J.S. Seminar given to the Dow Chemical Company on Sept. 7, 2000.

Self-healing Composites Using Embedded Microspheres: Fracture Toughness of Epoxy Matrix, Brown, E.N., International Congress of Theoretical and Applied Mechanics, Sept. 1, 2000, Chicago, IL.

Performance of Embedded Microspheres for Self-Healing Polymer Composites, E.N. Brown and N.R. Sottos, Society of Experimental Mechanics IX international Congress on Experimental Mechanics, June 7, 2000.

6.2 Air Force Lab Collaborations

We will begin a new project with AFRL/VSSV on the use of self-healing technology for cryogenic fuel tanks. These tanks are prone to leakage caused by microcracking as they cycle between cryogenic conditions and room temperature. We have teamed with a small company, CU Aerospace, to work on a phase-I STTR contract with AFRL to produce test articles suitable for cryo-testing at test facilities at Kirtland, AFB. This new project will commence in late September, 2002 and run for 6 months.

6.3 Transitions

We have supplied component materials to Wilson Composites, Goodyear Tire and Rubber Corporation, and Hempel Paints for evaluation and testing. In addition, we have signed xx non-disclosure agreements with companies interested in evaluating self-healing technology in their market applications. The University of Illinois' Office of Technology Management hosted a Technology Briefing on self-healing on July 23-24, 2002 at the Beckman Institute for Advanced Science and Technology in Urbana, IL. The briefing was attended by 21 companies and the university has begun active negotiations regarding licensing and development agreements.

7. Patent Disclosures

Multifunctional Composite Materials Using Embedded Microcapsules, Invention Disclosure TF00061, S.R. White, N.R. Sottos, P.H. Geubelle, J.S. Moore, S. Sriram, E. Brown, M. Kessler, and S. Viswanatan (Feb. 14, 2001)

8. Honors/Awards

Finalist Tech Museum of Innovation Award– Technology Benefiting Humanity (winners to be announced at the Awards Gala on Nov. 1, 2001 in San Jose, CA).

Prof. Scott White received the Xerox Award for Excellence in Research (2001)

Graduate Student Suresh Sriram was awarded 2nd place in the MRS Student Paper Competition (Boston, Dec. 1, 2000) for his presentation entitled: *Development of self-healing polymer composites.*

Graduate Student Michael Kessler received 2nd place SES Student Paper Competition (Univ. of South Carolina, Columbia SC, Oct. 23-25, 2000) for his presentation entitled: *Self-healing of delamination damage in woven glass/epoxy composites using embedded microcapsules.*

Graduate Student Eric Brown received the Henry L. Langhaar Graduate Award (2001) from the Department of Theoretical and Applied Mechanics.

Graduate Student Michael Kessler received the Louis J. Larson Graduate Award (2001) from the Department of Theoretical and Applied Mechanics. He also received a Beckman Fellowship (2001) to continue his graduate studies on self-healing composites.

S.R. White, N.R. Sottos, P.H. Geubelle, J.S. Moore, E.N. Brown, and M.R. Kessler received the American Society of Composites 2001 Best Paper Award in the Polymer Composites Division for their paper delivered at the 16th Annual Technical Conference, Sept. 9-12, 2001, Virginia Tech University, Blacksburg, VA.

Prof. Scott White was named a Willett Faculty Scholar (2002-05) for the College of Engineering, University of Illinois.

Prof. Nancy Sottos was named a University Scholar (2002-05) for the University of Illinois.

Prof. Nancy Sottos was given the University of Delaware Presidential Citation for Outstanding Achievement (2002).

Prof. Nancy Sottos received the University of Illinois Outstanding Engineering Advisor Award (2002).

Prof. Nancy Sottos was named to the Editorial Board of Composite Science and Technology (2002).

Graduate student Eric Brown won the American Society of Composites Graduate Scholarship Award (2002).

Graduate student Joe Rule holds a Hertz Foundation Fellowship (2001-05).

Graduate student Daniel Therriault holds a NATEQ Fellowship from the government of Quebec, Canada; a Nanoscale Science and Technology Fellowship (2002) from the University of Illinois; and a Carver Fellowship (2001) from the Univ. of Illinois